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RESEARCH MEMORANDUM

SOME STRESS-RUPTURE AND CREEP PROPERTIES OF MOLYBDENUM

DISILICIDE IN THE RANGE 1600° to 2000° F

By W. A. Maxwell

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Cleveland, Ohio~~CLASSIFICATION CHANGED~~

To

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~~Authority of J. W. Gandy, dated 12/1/53~~
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NATIONAL ADVISORY COMMITTEE
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RESEARCH MEMORANDUM

SOME STRESS-RUPTURE AND CREEP PROPERTIES OF MOLYBDENUM DISILICIDE

IN THE RANGE 1600° to 2000° F

By W. A. Maxwell

SUMMARY

An investigation of the stress-rupture and creep properties of hot-pressed molybdenum disilicide has produced the following results:

1. The stress-rupture properties can be summarized as follows:

Temperature (°F)	Stress (psi)	Time to rupture, (hr)	Creep rate (in./in./hr)
1600	35,000	107	0.000024
1800	20,000	224	.000028
1900	12,000	110	.00073
2000	10,000	85	.0018

2. The use of molybdenum disilicide above 1800° F may be limited by the creep rate rather than by the stress-rupture life.

3. The long-time strength of molybdenum disilicide at high temperatures is superior to that of the high-temperature alloys and titanium carbide ceramals.

4. A comparatively convenient and satisfactory method for creep and stress-rupture testing to 2000° F has been developed.

INTRODUCTION

Previous reports (references 1 to 4) have indicated the high short-time strength and outstanding oxidation resistance of molybdenum disilicide (MoSi_2) at 2000° F and above. The long-time strength of MoSi_2 in the range 1600° to 2000° F was surveyed to furnish stress-rupture and creep data for evaluating its use at these high temperatures.

The form of material selected was that which previous work (reference 3) had indicated to have superior long-time properties.

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2457

As reference 3 also had shown that the long-time properties could be varied considerably by changes in fabrication method, impurity content, etc., it was decided to investigate the best available form of the material in the knowledge that it did not necessarily represent the ultimate development of molybdenum disilicide. All specimens were prepared at the NACA Lewis laboratory.

An auxiliary objective of the investigation was the development of techniques for stress-rupture and creep evaluation to 2000° F. In view of the importance of long-time data for the evaluation of new materials, it appeared highly desirable to develop a convenient method of testing brittle materials to at least 2000° F by methods sufficiently similar to the conventional long-time evaluations of alloys to permit correlation of the results. Self-aligning metallic grips and other improvements were therefore developed for long-time evaluations.

APPARATUS AND PROCEDURE

Design of apparatus. - Long-time testing of MoSi_2 at temperatures up to 2000° F presents several problems, the most important of which is that of aligning the specimen so as to prevent failure in bending. In addition, it appeared desirable to develop a method which would require a small, easily prepared specimen and easily fabricated holders, or grips, for the specimen.

Two methods appeared feasible for attaining alignment. The first called for careful prealignment of the specimen in precisely machined and carefully adjusted grips. Any misalignment could be accurately measured at room temperature by the use of strain gages, and operations could be carried out carefully in a manner which would tend to maintain the alignment during heating and loading. Experiments with the pre-alignment method with strain gages showed the method to be troublesome and laborious; a further disadvantage was that alignment would not necessarily be maintained during actual testing.

The second method consists in using grips capable of deforming plastically to an extent sufficient to permit the specimen to align itself under load. The plastic seating of the specimen could be accomplished by the use of either (1) a comparatively long specimen held by grips outside the furnace and seated in a material plastic at room temperature or considerably below the test temperature, which would leave only the gage length of the specimen at temperature, or (2) a shorter specimen completely in the furnace and held in grips of a material having sufficient plasticity at or near the test temperature to assure alignment.

The method employing a long specimen held outside the hot zone (references 5 and 6) appeared to offer considerable promise for temper-

atures above 2000° F. However, this method requires a longer and more difficultly formed specimen. This is a marked disadvantage for experimental materials which are available only in laboratory quantities and must be fabricated by laboratory methods.

2457 The method developed here employs a small and easily fabricated specimen and closely resembles conventional evaluation methods. Only the holders present a problem. Ceramic holders can be fabricated only with difficulty and would require special deformable inserts. The high-temperature alloys such as Inconel X can be machined, but their creep rates at 2000° F are high. It appeared possible to take advantage of the plasticity of some of the high-temperature alloys above their normal temperatures, so that the creep of the alloy could furnish the plastic deformation necessary for alinement. It was hoped that by proper design the stress and consequent creep rates of alloy holders could be reduced to the extent that a given set of holders could be used for several evaluations under reasonably high stresses at temperatures to 2000° F. Such a method was indicated in reference 2 to be feasible. The holders, inserts, and specimen shape were adapted from those developed at the NACA Lewis laboratory for high-temperature tensile testing (reference 7).

Apparatus. - The grip arrangement used is shown in figure 1 and consists of a set of inserts in contact with the specimen, holders which support the inserts and are connected to the pull rods through a heavy pin, and the air-cooled pull rods. Air cooling was employed to minimize creep in the pull rods and deformation of the pins. The cooling effect is probably small below the pin area. All parts were machined of annealed Inconel X. The arrangement was set within a conventional platinum-wound furnace and mounted on a stress-rupture machine as shown in figure 2.

Because the rather extensive creep of the Inconel is added to the specimen creep, the total elongation of the system is excessive and would result in undesirable effects such as movement of the specimen from the center of the furnace. The specimen might also be subject to bending forces. The elongation is sufficient so that the loading beam would be lowered to its rest point, and the load would thereby be removed. An arrangement was therefore necessary to provide an automatic take-up of the elongation and maintain a level beam. A device developed for this purpose by Mr. Paul F. Sikora of the NACA Lewis laboratory consists of a motor-driven gear-train mechanism which drives a guided screw connected to the lower pull rod. The mechanism is actuated by upper- and lower-limit switches on the beam. The switches can be arranged to compensate for elongations as small as 0.0003 inches. Because the screw is guided and the motion slow (approximately 0.003 in./min), the specimen is not subject to shock.

Specimen creep is measured by means of platinum extensometers of the type described in reference 8. These are attached to the specimen with a silicate-alumina cement as shown in figure 1. A wire, not in contact with the specimen, is used to hold the extensometer in place while the cement sets. Measurements of creep are made with a telescope, as shown in figure 2, to an accuracy of 0.001 centimeter. As the beam is maintained level and only one viewing window is available, only one extensometer is used on each specimen. At temperatures of 1800° F and above, the light of the furnace is sufficient for convenient reading of the extensometers. At 1600° F the fine lines on the platinum are distinguished with difficulty. It was discovered that if a quartz rod was inserted obliquely through the furnace wall, the light of a flash-light bulb at the outside end of the rod was sufficient to illuminate the inside of the furnace.

Temperature was measured by three thermocouples placed at the top, bottom, and center of the gage length. The center temperature, which was used for control purposes, was automatically recorded. Temperature was controlled by a Celestray controller.

Evaluation procedure. - Specimens were mounted in the holders as indicated by figure 1. Specimen-to-metal contact was prevented by coating the contact portions of the specimen with a suspension of cerium oxide in water, which is necessary to facilitate removal of specimens. A preload of 10 pounds (equivalent to 2000 psi stress in the average specimen) was then applied. After the furnace position was adjusted, heat was applied and cooling air was admitted to the grips at a rate such that when the specimen was at test temperature the air left the grips at a temperature of approximately 550° F. The specimen was "soaked" for one hour under the preload stress at the test temperature and the full load then applied slowly enough to allow the gear-train mechanism to maintain the beam level. Extensometer readings were taken three times during the working day. For material of limited creep, readings were omitted on week ends.

Preparation of specimens. - Previous research (reference 3) had indicated the superior creep properties as determined by the flexure-creep test of MoSi_2 of large grain size and comparatively low oxygen content fabricated by hot-pressing. Specimens were prepared as described in detail therein.

The powder particle size analysis as obtained microscopically was:

Particle size (microns)	Percent of particles
Greater than 15	1
Less than 15, greater than 11	1
Less than 11, greater than 7	3
Less than 7, greater than 3	11
Less than 3, greater than 1	33
Less than 1	51

Bars were prepared by hot-pressing the powder in graphite dies to form a bar $\frac{1}{2}$ by $\frac{1}{2}$ by $3\frac{1}{2}$ inches. The pressure of 2400 pounds per square inch was applied on the $\frac{1}{2}$ - by $3\frac{1}{2}$ -inch face. The dies were heated by induction to 3100° F in 30 minutes and the pressure and temperature maintained for an additional 30-minute period. Bars so produced had a density of 6.10 (approximately 98 percent of the theoretical density of 6.24 as given in reference 1) and a representative chemical analysis of: molybdenum, 62.15 percent; silicon, 34.79 percent; carbon, 0.34 percent; oxygen, 0.42 percent; iron, 0.82 percent; and nitrogen, 0.03 percent.

Tensile bars similar in composition to those previously evaluated in flexure-creep (reference 3) were used in two stress-rupture tests. The particle size analysis of this powder was:

Particle size (microns)	Percent of particles
Less than 6	100
Less than 2	98

These bars were fabricated by cold-pressing at 40,000 pounds per square inch by the hydropress method and sintered in vacuum at 2550 \pm 50° F for 2 hours, as described in detail in reference 3. A representative analysis of the finished bars was: molybdenum, 62.23 percent; silicon, 32.70 percent; carbon, 0.29 percent; oxygen, 1.2 percent; metallic impurities, present only as traces. The average density of these bars was 5.95 grams per milliliter.

Test specimens were ground from the bars with diamond abrasives to the shape shown in figure 3. Approximately 90 grams of powder was required for the bars; the finished specimens weighed 30 grams. All specimens were examined for flaws by a dye-penetration (Dy-chek) method.

Metallographic examination. - Broken specimens were cut so as to give two samples to provide cross sections both parallel to the failure plane and perpendicular to it. Samples were prepared by conventional methods with diamond abrasives and polishing powders.

RESULTS AND DISCUSSION

Comments on the Method

The apparatus and equipment discussed permit the testing of brittle materials with a degree of convenience approaching that of the testing of alloys. That the method provides good alignment of the specimen is indicated by the consistency of the data and the fact that no specimen was broken except as a test result. All specimens failed in the gage length and, with only one exception, at or quite close to the center. The method employed for the measurement of creep is satisfactory. The life of the holder is evidently quite long under the stresses used. At 2000° F, appreciably higher stresses may result in severe damage to, but not failure of the holders, a condition which would permit the completion of any given test. Temperatures above 2000° F are not recommended for the holders described.

Stress-Rupture and Creep Behavior

Stress-rupture results are shown in table I and plotted for three temperatures on figure 4. It will be seen from the graph that the plotted points show a limited scatter, a scatter which compares quite favorably with that found for commercial alloys produced in tonnage quantities. It therefore appeared justifiable, even with the limited number of points presented, to draw in the lines on the graph as representing the trends for the stress-rupture behavior of the material. The lines for 1900° and 2000° F have been drawn parallel to the 1800° F line at fixed distances from it. Two results for an evaluation at 1600° F are listed in the table.

The data given in table I indicate that at temperatures above 1800° F the use of molybdenum disilicide may be limited by the creep rate rather than by the stress-rupture life. For comparison purposes, the times required for a 3-percent extension by creep have been shown in table I. Creep curves are given on figure 5 in inches per inch for various stresses and temperatures. The slope of these curves rises markedly for temperatures above 1800° F. First-stage creep, if any, apparently occurs during loading and is masked by the loading method. Definite third-stage creep is found for all specimens.

2457

A comparison of hot-pressed molybdenum disilicide with other high-temperature materials is given in table II. At 1800° F the 100-hour strength of MoSi₂ is far superior to that of the comparison materials. Creep does not appear to be a limiting factor for the silicide at this temperature. Above 1800° F the silicide is surpassed in strength only by the alumina-chromium cermet. Although the long-time, high-temperature strength of molybdenum disilicide is outstanding, there is some evidence (reference 9) that the silicide requires further development to improve its thermal shock characteristics before it can be used as a turbine-blade material.

The two specimens of fine-grain, high-oxygen, sintered material, as shown by comparison of their reductions in area and life with those of hot-pressed specimens under the same stress, are seen to deform plastically to a far greater extent than does the larger-grain, lower-oxygen material. This result confirms at least qualitatively the previously reported flexure-creep evaluation (reference 3). The extent to which the fine-grain material can deform without failure is shown on the photograph in figure 6 in which a reassembled specimen is compared with its original form. The fact that a specimen could undergo a 50-percent reduction in area and elongate as shown in the photograph suggests that the previously reported high-temperature plasticity of molybdenum disilicide (references 1 and 3) may be due, to at least some extent, to the high creep rate of the material.

Structural Changes

Metallographic evaluation has revealed several aspects of damage to molybdenum disilicide under prolonged stress at high temperature. Possibly the most noticeable change in the material caused by the evaluation conditions was indicated by the difficulty with which specimens of failed material were polished. The untested material was readily polished to a good surface, while a usable surface on the failed specimens was attained only with the greatest difficulty. This deterioration is shown in the photomicrograph (figs. 7 and 8) by a comparison of figure 7(a), the material as prepared, with the structures found after evaluation. The presence of a greatly increased number of voids is evident in all the failed specimens. The small circular voids are probably formed by the removal of material during preparation; it is this tearing out of material which is responsible for the difficulties encountered in polishing. The fact that tearing out of material occurs would indicate a deterioration in the bonding of the torn-out particles. In the sections perpendicular to the fracture plane (fig. 7(c), 8(b), and 8(c)), cracks transverse to the direction of applied stress are quite evident. This cracking is more marked in the specimens evaluated at the higher temperatures. In most cases these cracks appear to be caused by

separation of the grains. From a study of the failed specimens, intergranular separation appears to be an important factor in the stress-rupture failure of molybdenum disilicide, a phenomenon common with alloys.

SUMMARY OF RESULTS

An investigation of the stress-rupture and creep properties of hot-pressed molybdenum disilicide has produced the following results:

1. The stress-rupture properties can be summarized as follows:

Temperature OF	Stress (psi)	Time to rupture, (hr)	Creep rate (in./in./hr)
1600	35,000	107	0.000024
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2. The use of molybdenum disilicide above 1800° F may be limited by the creep rate rather than by the stress-rupture life.

3. The long-time strength of molybdenum disilicide at high temperatures is superior to that of the high-temperature alloys and titanium carbide ceramics.

4. A comparatively convenient and satisfactory method for creep and stress-rupture testing to 2000° F has been developed.

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TABLE I - SOME STRESS-RUPTURE AND CREEP PROPERTIES OF HOT-PRESSED

LARGE-GRAIN MOLYBDENUM DISILICIDE



Temperature	Stress (psi)	Time to rupture (hr)	Creep rate, second stage, (in./in./hr)	Time for 3-percent elongation (hr)	Reduction in area (percent)
1600	25,000 35,000	plus 2,000 ^a 107	0.000015 .000024	2000 failure ^b	1.6 0
1800	10,000 15,000 20,000 10,000 ^d	1389 424 224 plus 843 ^c	.000028 .000096	failure ^b 312	7.8 3.1 1.6 7.8
1900	10,000 12,000 12,000 15,000	320 103 110 87	----- .000078 .000073 -----	----- 38 42 -----	3.1 7.8 7.8 3.2
2000	7,500 10,000 15,000 7,500 ^d	152 85 22.5 81.2	----- .0018 ----- -----	----- 18 ----- -----	9.4 9.4 7.1 50.3

^aTest discontinued and specimen loaded in 1000-psi increments to failure at 29,000 psi.

^bFailure occurred before 3 percent elongation was attained.

^cTest discontinued and specimen loaded to 33,000 psi without failure.

^dFine-grain, sintered MoSi₂.

TABLE II - COMPARATIVE STRESS-RUPTURE STRENGTHS OF REPRESENTATIVE
HIGH-TEMPERATURE MATERIALS

[Stress in psi for 100-hour life]



Test temper- ature	Cemented carbide ^a		Cast alloys		Forged alloy S-816 ^c	Cermets ^d A-12-b	Molyb- denum ^e	Molyb- denum ^f disil- icide
	K-151A	K-152B	x-40 ^b	Haynes Stellite no. 21 ^c				
1600	20,000	15,000	21,000	16,700	14,500			35,000
1800	12,000	4,500	11,300	9,000	5,300	16,300	19,300	29,000
1900	8,000							13,500
2000	4,000					13,500		8,500
2200						12,200		

^a"Kentanium" bonded titanium carbide (reference 10).

^bReference 11.

^cReference 12.

^dComposition of cermet, 30 percent chromium, 70 percent alumina
(reference 13).

^eCold-pressed, sintered molybdenum (reference 14).

^fData from figure 4.

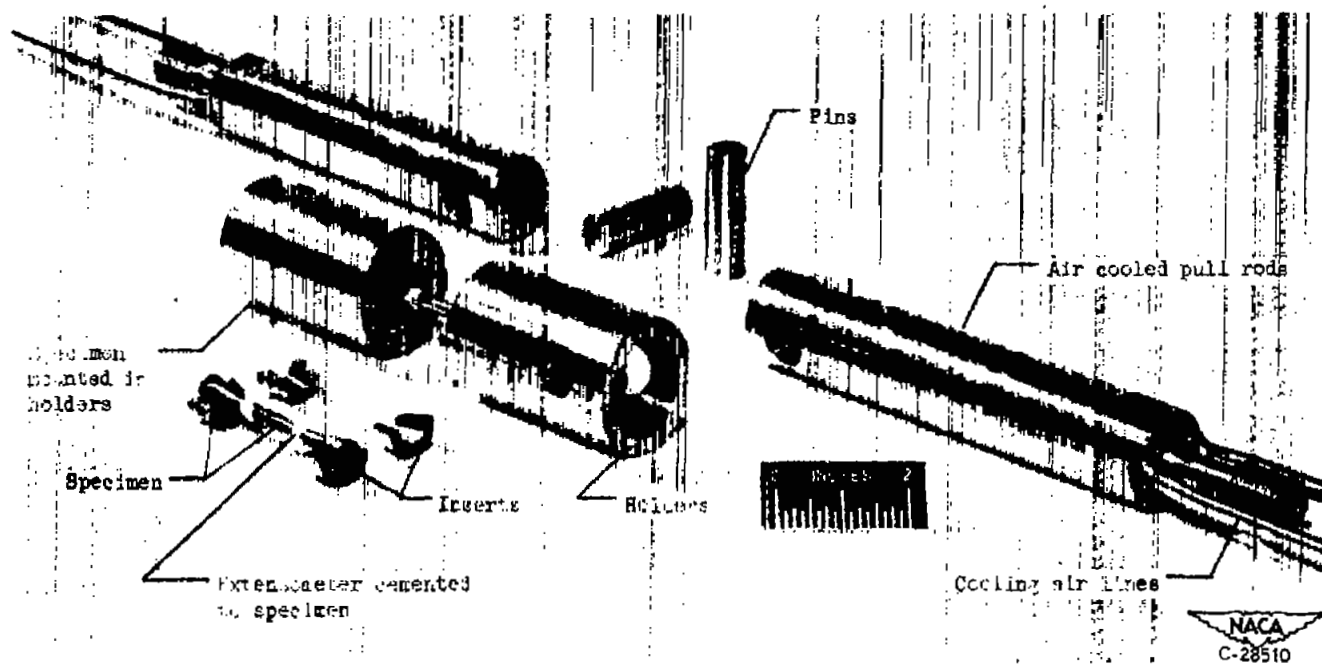


Figure 1. - Specimen grip assembly for long-time evaluations to 2000° F.

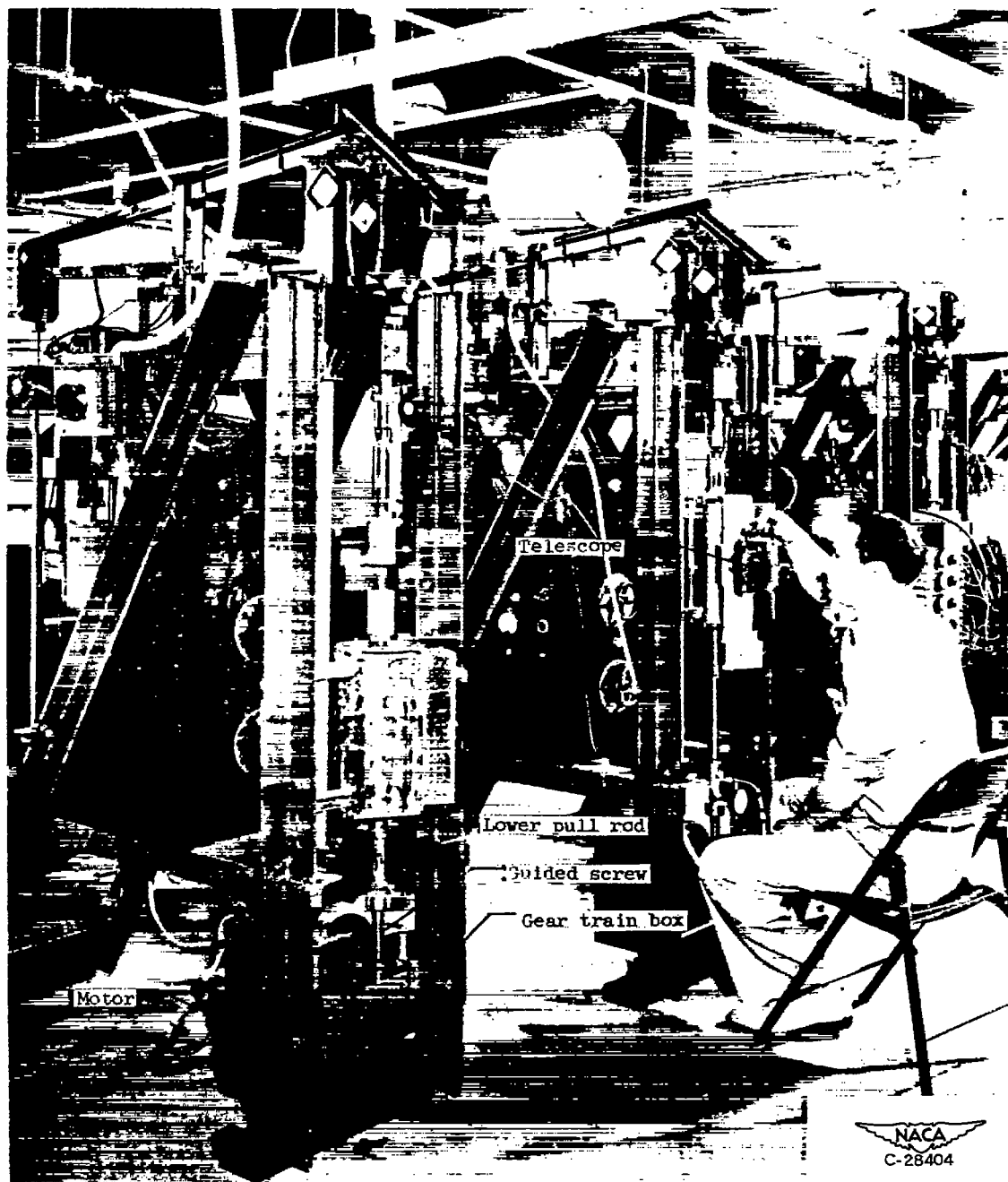


Figure 2. - High-temperature stress-rupture machines. Left, specimens in place and pre-loaded; right, evaluation in progress, telescope in place for creep measurement.

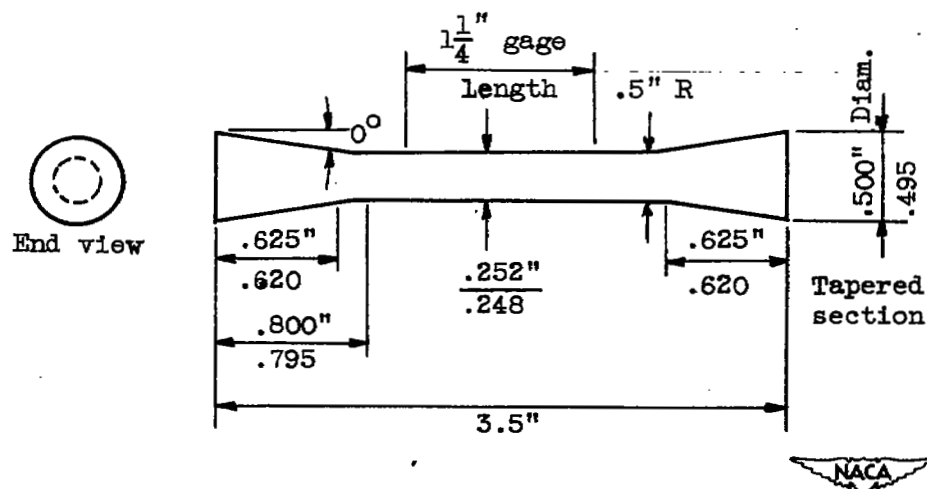


Figure 3. - Specimen used for creep and stress-rupture tests of molybdenum disilicide.

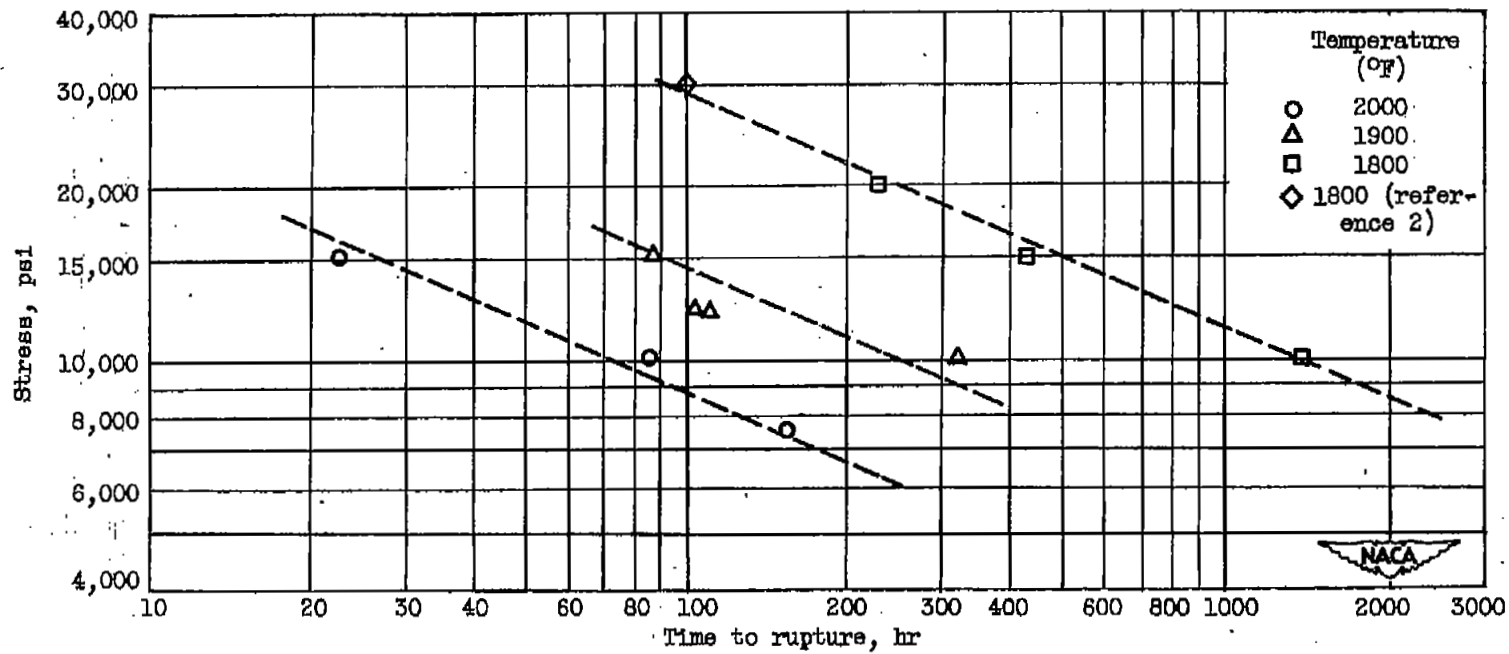


Figure 4. - Stress-rupture curves for molybdenum disilicide.

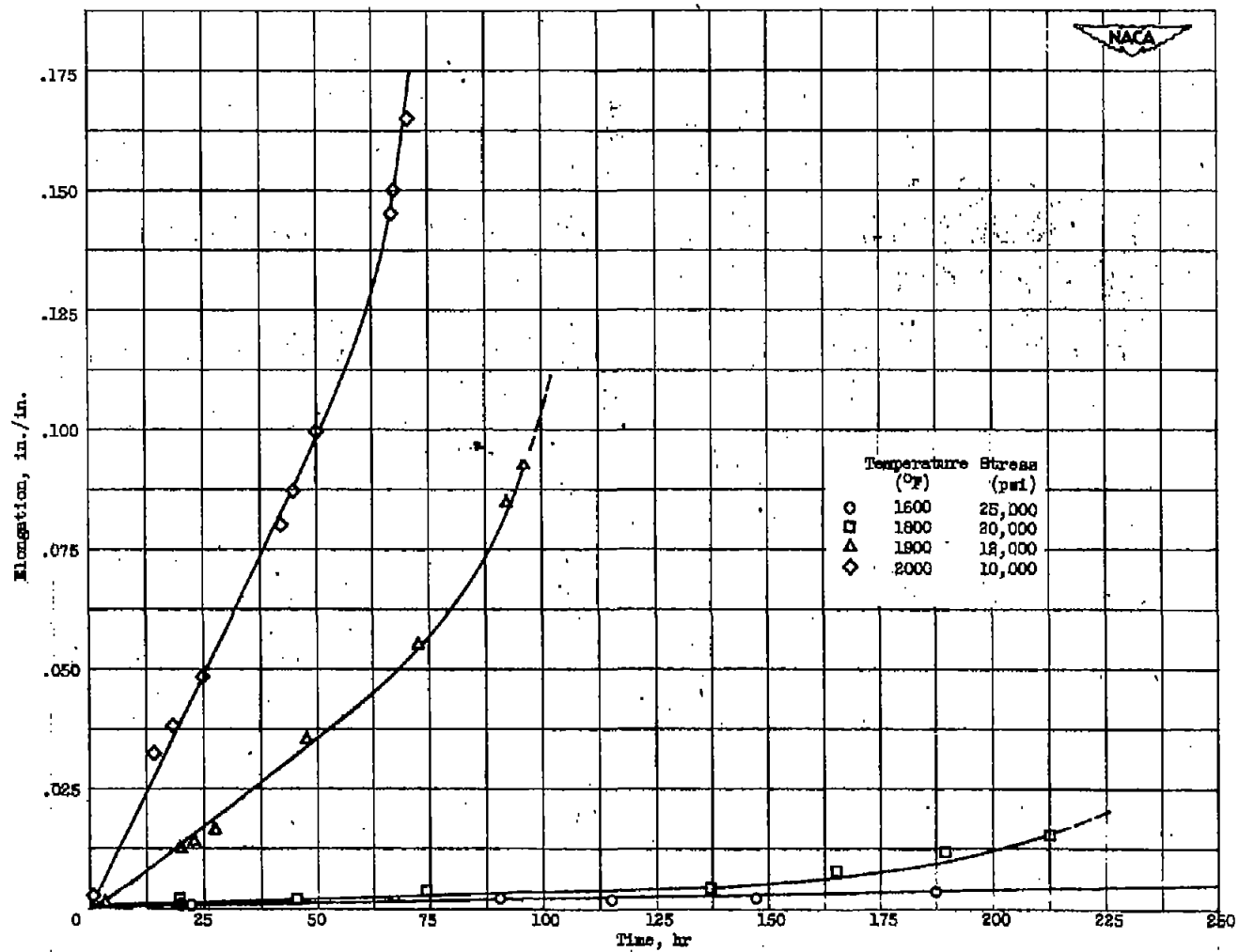


Figure 5. - Creep behavior of hot-pressed molybdenum disilicide.

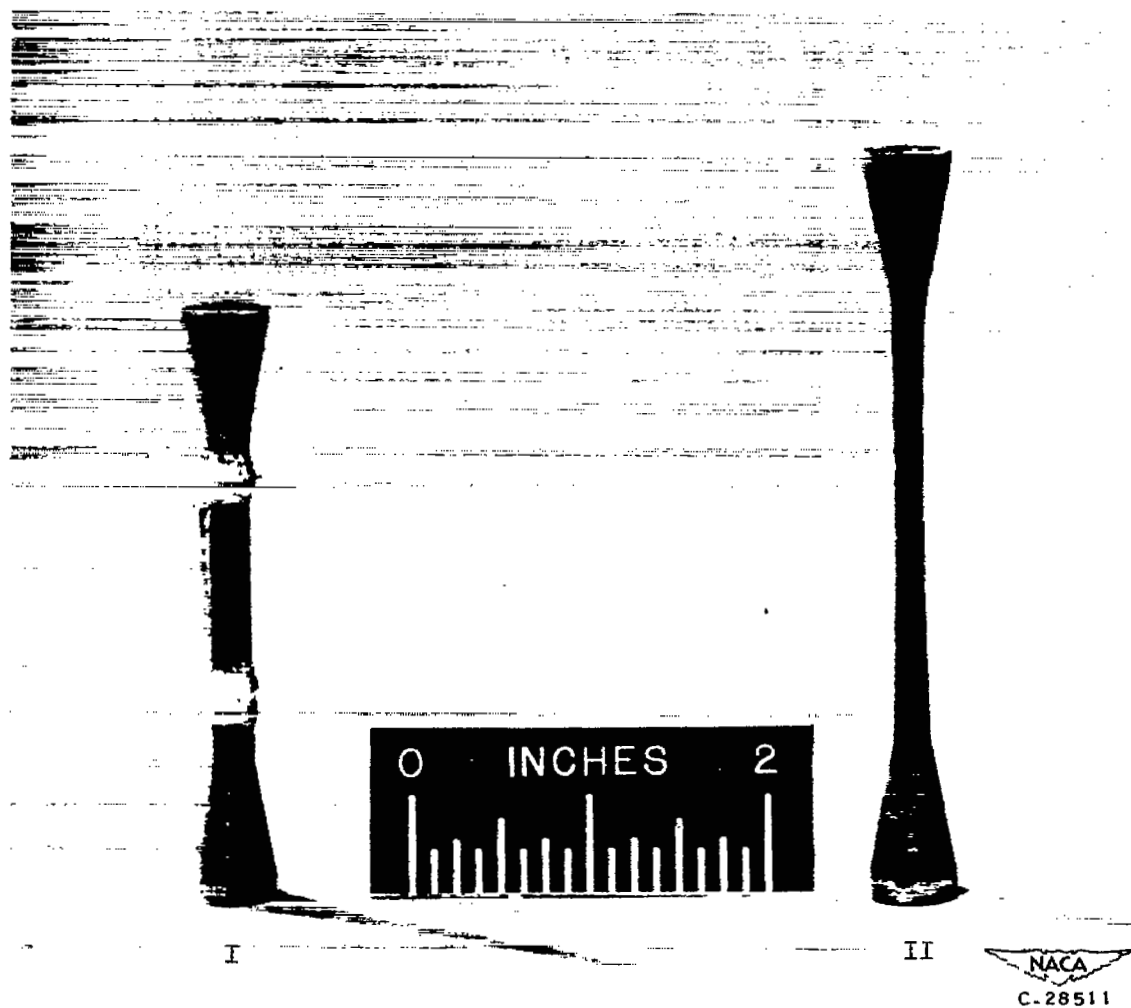
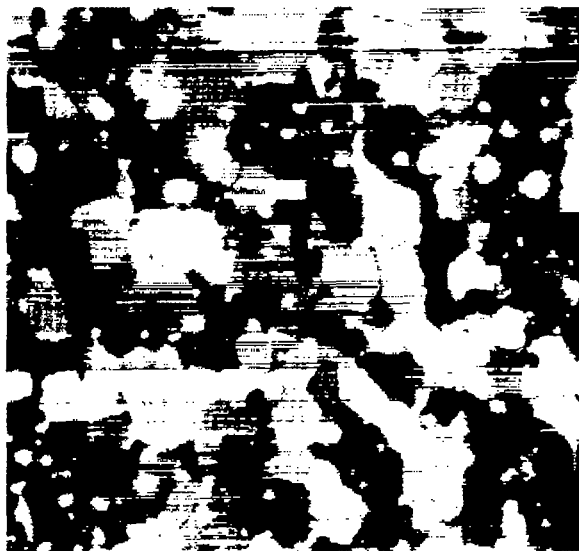
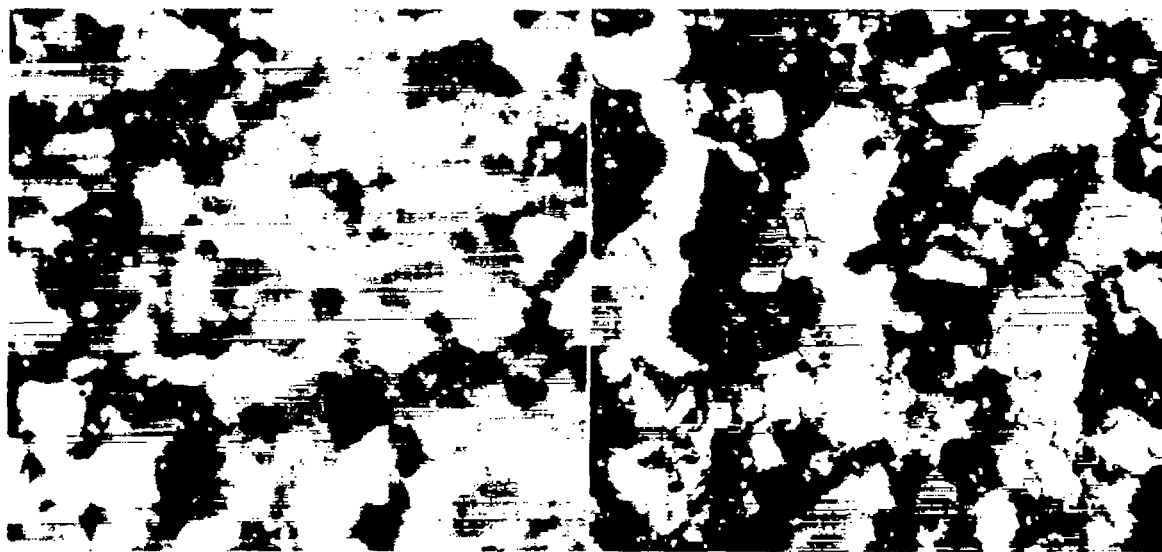


Figure 6. - Sintered, fine-grain molybdenum disilicide. I, specimen before testing; II, specimen reassembled after 81 hours at 2000° F and 7500 pounds per square inch.



(a) Hot-pressed material as prepared before evaluation in stress-rupture.

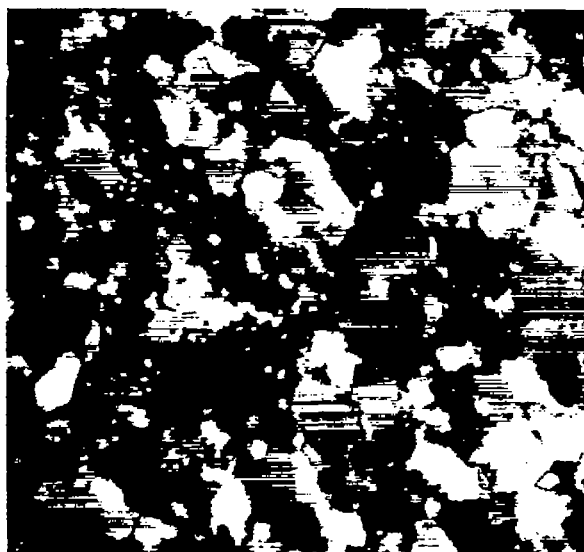


→ Direction of stress

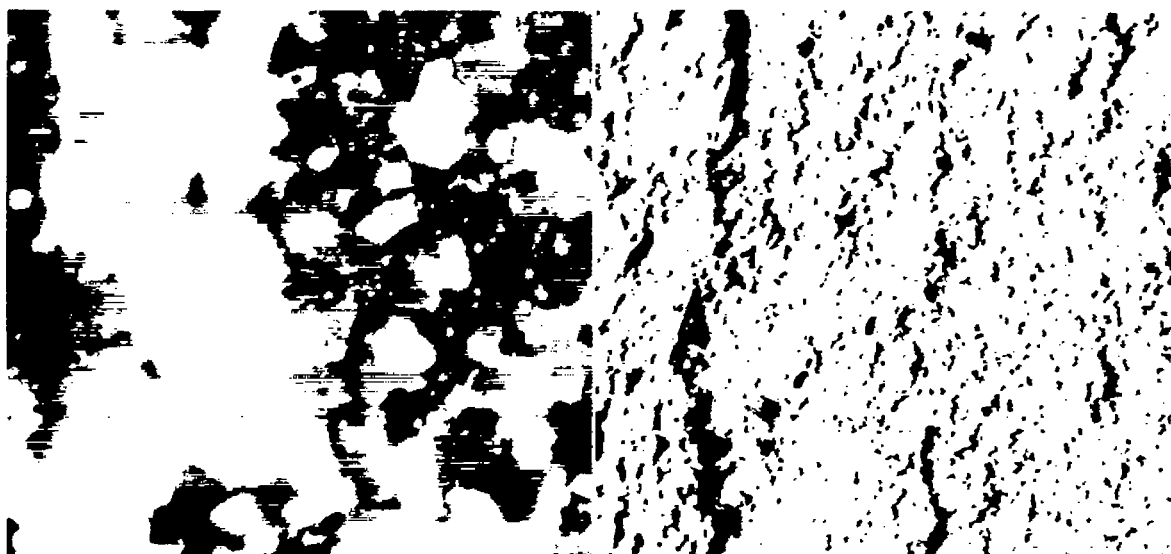
(b) Specimen evaluated at 1800° F and 10,000 pounds per square inch after failure at 1389 hours. Section parallel to fracture plane.

(c) Specimen identical with that of figure 7(b), but showing section perpendicular to fracture plane.

Figure 7. - Photomicrographs of molybdenum disilicide; X1000; polarized light; unetched.



(a) Section parallel to failure plane; X1000; polarized light; unetched.



→ Direction of stress

→ Direction of stress

(b) Section perpendicular to failure plane; X1000; polarized light; unetched.

(c) Section perpendicular to failure plane; X250; normal light; unetched.

Figure 8. - Specimens of molybdenum disilicide evaluated at 2000° F and 7500 pounds per square inch.

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